











Leaders in Environmental Testing

### Emerging Technologies – Toxic Organics











**Advances in High-Volume Sampling** and Trace Analysis of Persistent **Organic Pollutants** 

# Newark Bay Estuary Participants



- **► United States Geological Survey (USGS-NJ)**
- ➤ New Jersey Department of Environmental Protection (NJDEP)
- >Stevens Institute Rutgers University
- **▶** Severn Trent Laboratories
- **▶** Battelle Environmental Divisions

# New Jersey Toxic Sediment Reduction Program

- Determine sources and concentrations of organic contaminants in the Newark Bay and Hudson River Estuary system.
- Detect as many target compound as possible.
- Obtain samples with target compounds in a range of magnitudes above field blanks and lab blanks.
- >> High-volume multi-stage sampling train. Toxic Organics Platform Sampler (TOPS).

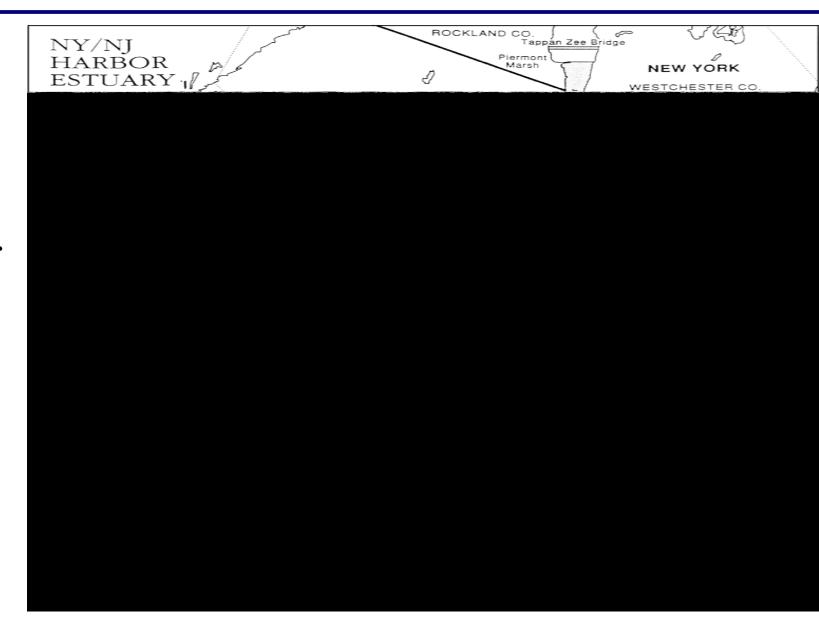
# Persistent Organic Pollutants (POPs) Studied

- Polychlorinated Dioxins and Furans (PCDDs/PCDFs) (17 analytes)
- Polychlorinated Biphenyls (PCBs) (113 analytes)
- Organochlorine Pesticides (OCPs) (24 analytes)
- Polynuclear Aromatic Hydrocarbons (PAHs) (27 analytes)

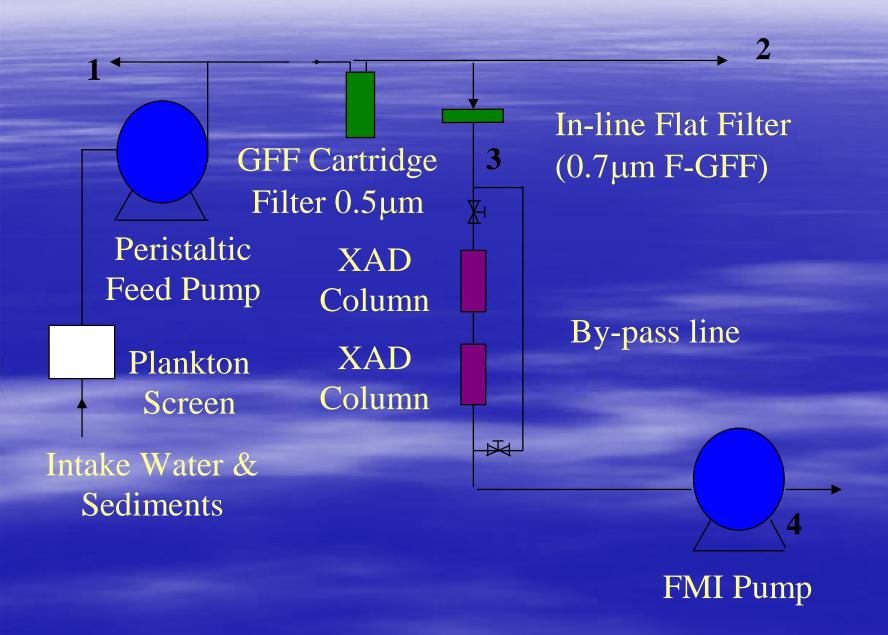
### New York / New Jersey Harbor Estuary

# **Sampling locations:**

- heads of tide,
- combined sewer outfalls
- mixed pools.



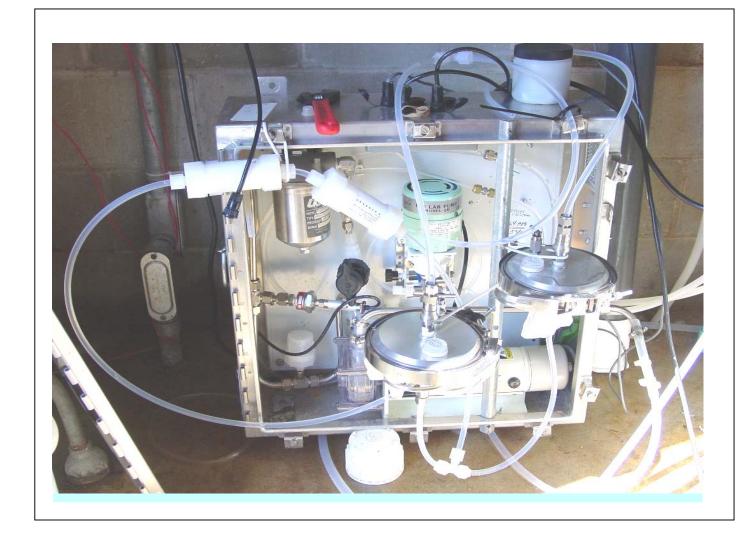
# Modified TOPS Sampler



#### **TOPS Sampler**

# Large volume sediment/water phase sampler using filters and XAD resin columns





## Filters

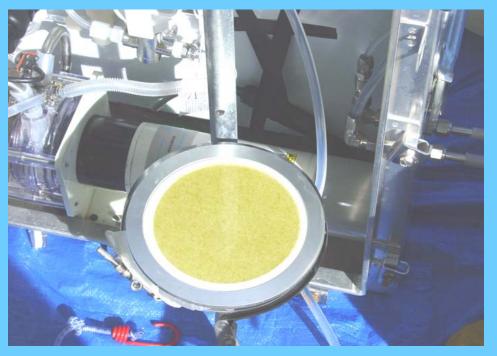


Flat GF/F

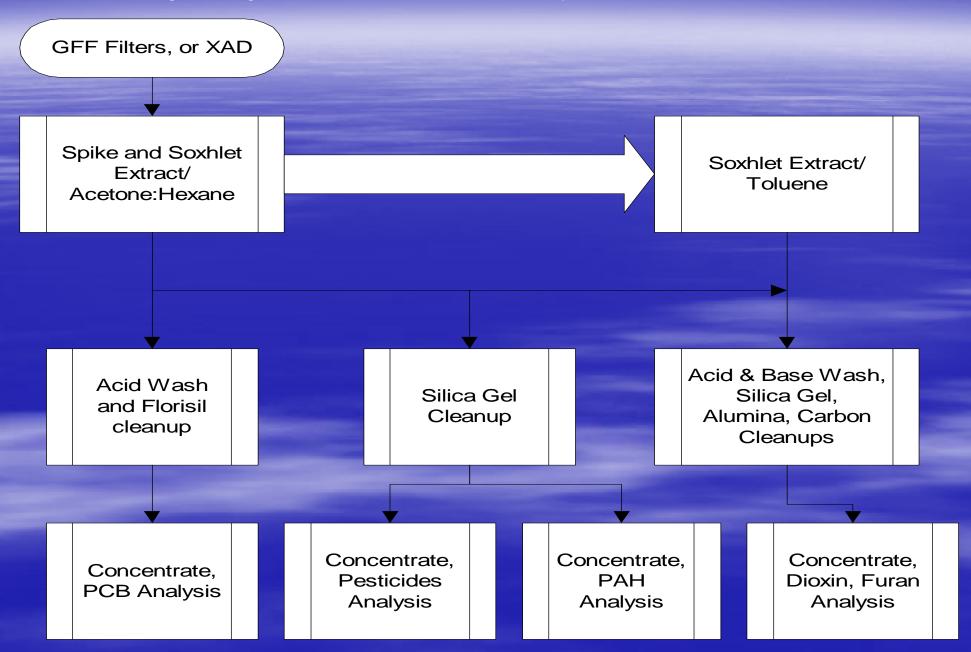
142 mm diameter

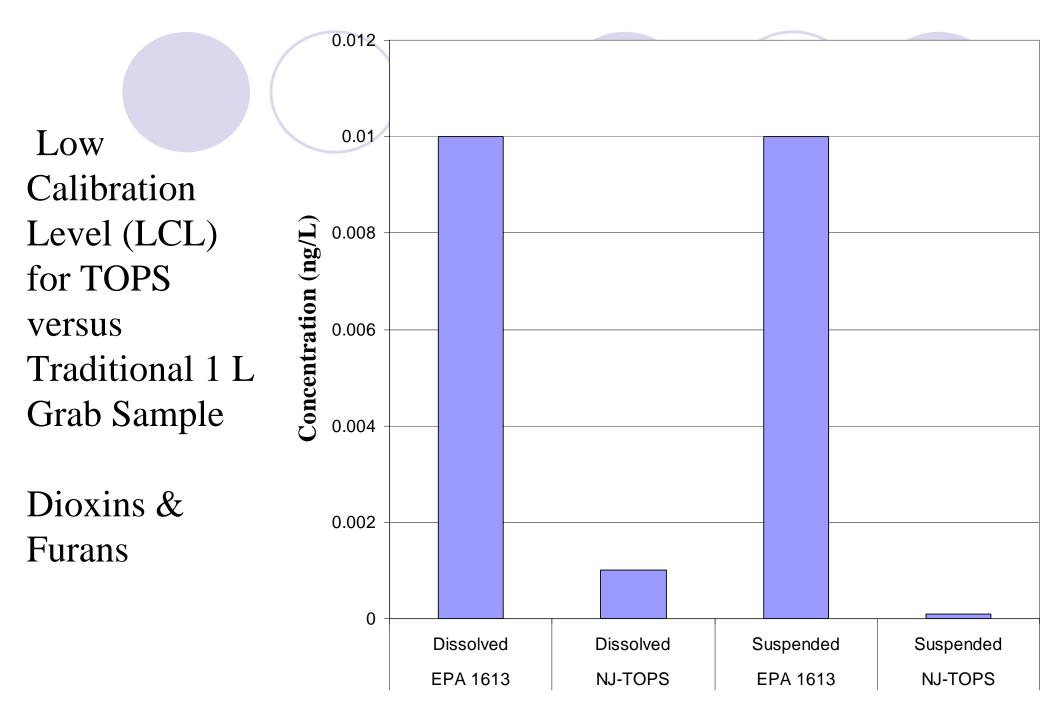
0.7 micron,
nominal pore
size

GFF Canister
'4" in length
0.5 micron,
nominal pore
size



#### Laboratory Analysis – Extraction, Cleanup, Concentration, GC/MS

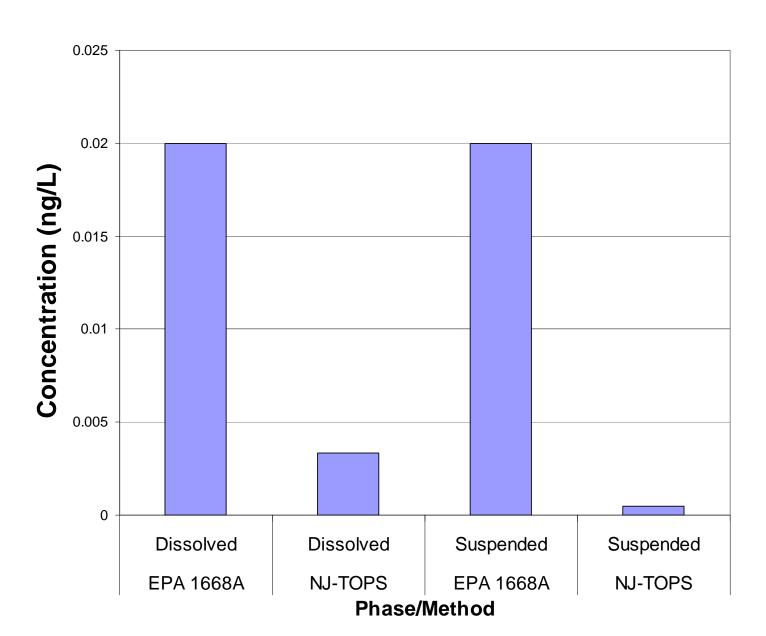


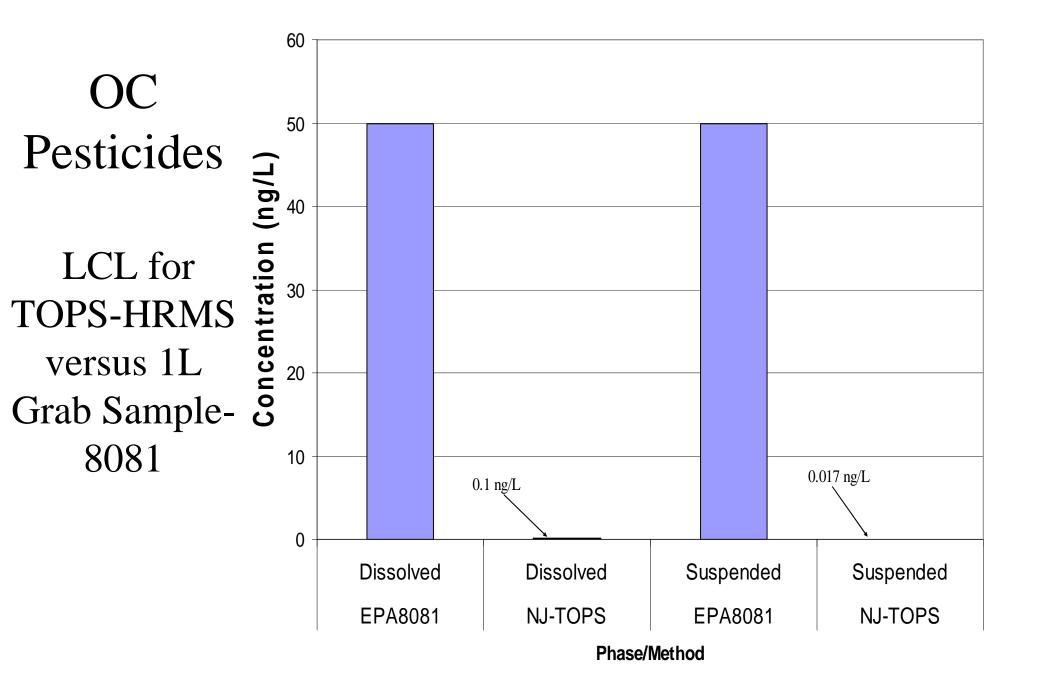


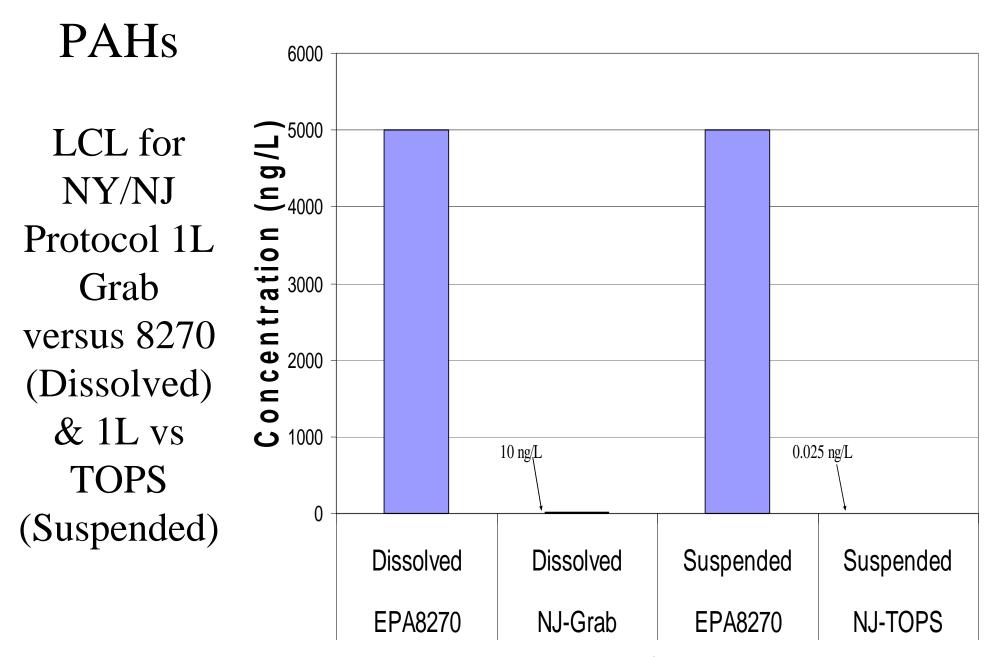
Phase/Method



LCL for TOPS versus 1L Grab Sample







Phase/Method

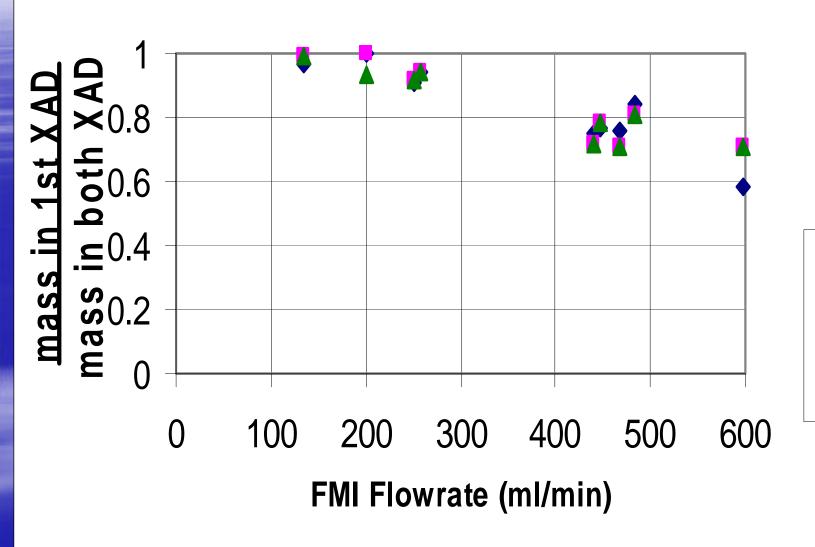
### Recovery of NIST SRMs from XAD resin Typical time between spiking and analysis = 7 to 30 days

Analyte Class	Spiking Medium	Spike Levels ng/sample	Certified Analytes	Lowest Recovery	Highest Recovery	Average Recovery
OC						
Pesticides	XAD	1-5	14	66	130	88.4
PCBs	XAD	20-50	23	82	157	102
Dioxins	XAD	1	1		111	

#### **Recoveries of NIST PAH SRM from Water**

PAHs	Water	100-500	22	78	133	101
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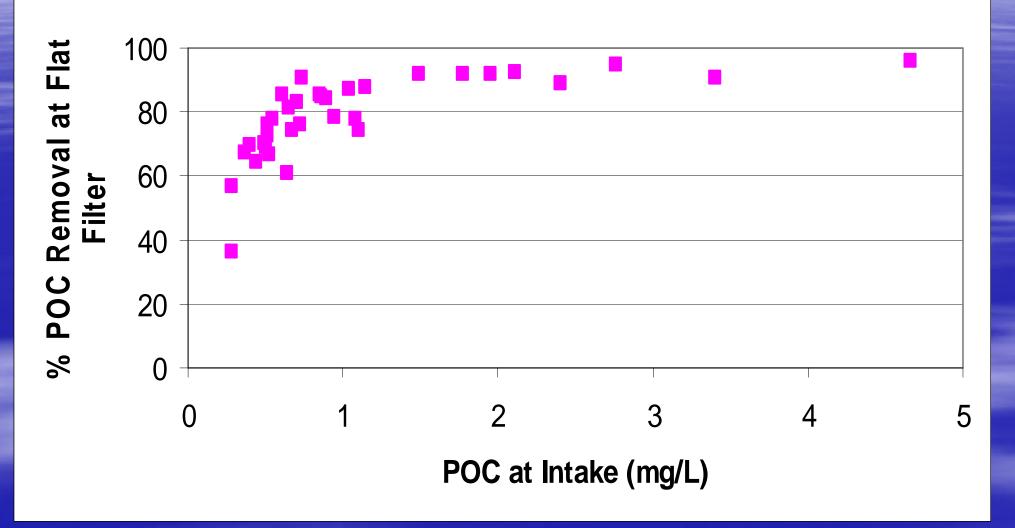
### Pesticide Data FMI flowrate vs. Breakthrough



- Lindane
- Dieldrin
- ▲ Alpha-BHC



#### **POC Removal at Flat Filter**





How low can we measure? Example - PCB Congener EDLs (EDL = Concentration associated with a peak at 2.5 X noise, during analysis.)

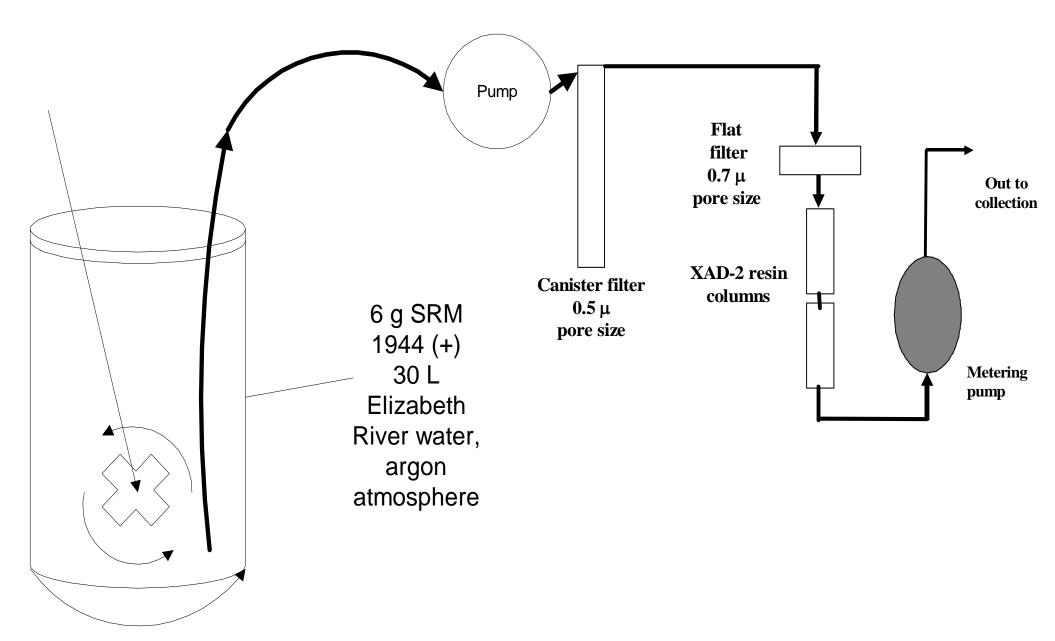
Range of values observed in this work:

- $\triangleright$  Dissolved (XAD) = 15 to 450 pg (0.3 to 9 pg/L for 50 L sample)
- Filters = 25 to 550 pg (8 to 180 pg/g for 3 gram sample) For a 500 L sample at 6 mg/L, 0.016 to 0.36 pg/L

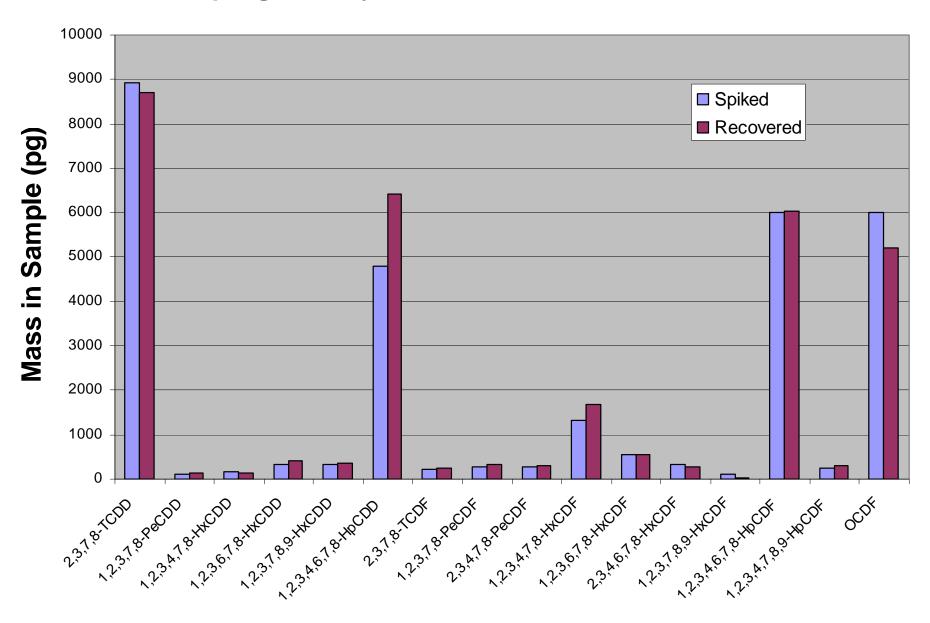
This assumes that 100% of contaminants entering the sampler are trapped by the filters and XAD, AND recovered through analysis.

An evaluation of sampling & analysis validity was performed by spiking river water with NIST sediment and liquid SRMs.

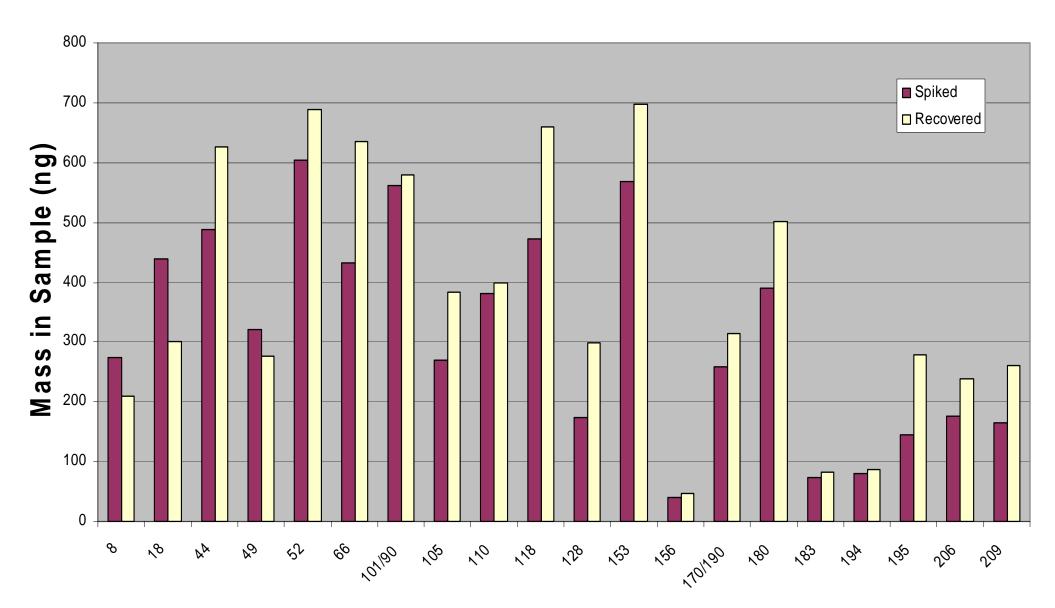
# Validation Study - Apparatus



#### Sampling & Analysis Validation - Dioxins & Furans



### **Sampling & Analysis Validation PCBs**



**Congener (BZ Number)** 



### Summary



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- Operating limits were determined for flow rate, filter configuration, minimum POC, XAD bed volume.
- Calibration range for all compound classes were reduced by 1-4 orders of magnitude below EPA Methods.
- •Measured detection limits (EDLs) have been demonstrated at ppq and sub-ppq levels for POPs.
- Laboratory analysis precision and recovery have been demonstrated by SRM analysis. (PAHs, PCBs, OCPs, TCDD).
- Sampling and analysis accuracy has been demonstrated by SRM analysis (PCDD/Fs, PCBs). PAH data is in progress.
- ■TOPs met program goals for PCDD/F, PCB, PAH (sediments). OCP data not yet evaluated.



# Acknowledgements





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- ➤ Timothy Wilson, Jennifer Bonin– USGS, Trenton, NJ
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- **▶** Joel Pecchioli New Jersey DEP, Trenton, NJ
- ➤ Tsian Liang Su Stevens Institute Rutgers University
- ➤ Simon Litten, Larry Bailey New York DEC, Albany NY











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